FIREFLOODING CHANGES IN ATHABASCA BITUMEN AND WATER PROPERTIES

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INTRODUCTION

Fireflooding is the process of igniting the hydrocarbons in the formation at the site of an injection well; this is followed by propagation of the combustion front through the reservoir to producing wells. Combustion is maintained by the injection of air and as the fire-front moves through the reservoir, it vaporizes oil and the formation water. These are moved ahead of the front in a gas phase, then condensed in cooler portions of the reservoir and eventually produced from a production well. The fuel for combustion is supplied by the heavy residual material which is not vaporized by the fire-front. This material is generally referred to as coke and is deposited on the rock matrix.

Combustion can proceed in two directions: in the most commonly used process, the combustion zone advances in the same direction as the air flow and is known as "forward combustion"; in the second method, the combustion front advances in the direction opposite to the flow of air and is called "reverse combustion". Forward combustion burns the least desirable fraction of the oil, leaves a clean formation behind and is an efficient heat generating process. Its main drawback is that there must be sufficient mobility for the vaporized oil and water to be produced after they have condensed ahead of the firefront. In reverse combustion, the produced liquids are produced through the heated portion of the reservoir. The fuel for this process is an intermediate fraction of the original oil and the coke remains in the matrix. The process also produces crude oil which contains more oxygen compounds than crude produced with forward combustion. Because spontaneous ignition can occur the process is difficult to control.

In the forward combustion process, water has been injected with the air to remove heat from the hot rocks and to reduce the amount of air required. A number of investigations of different types of wet combustion have been made (1-3). These investigators have classifications for this process according to the amount of water being injected. In this paper we will use Burger and Sahuguet's (1) nomenclature. They classified the types of wet combustion into normal, incomplete and super wet. In normal wet combustion the water evaporation front is behind the firefront and all the coke is burned. In incomplete combustion the water evaporation front is behind the combustion zone but not all the coke is burned. In both of these processes superheated steam passes through the firefront. Super wet combustion takes place when enough water enters the combustion zone to cause the disappearance of the peak combustion temperature.

The object of this study is to present the data from the analysis of the effluent of a dry combustion test and a wet combustion test performed on oil sands from the Athabasca Oil Sands deposit. The paper gives a brief description of the apparatus used to run the tests, as well as a description of the analytical methods used to do the effluent analysis and a discussion of the results obtained.

EXPERIMENTAL APPARATUS

A schematic diagram of the combustion tube is shown in Figure 1. The combustion tube is constructed from a 1.83 meter length of 10.2 cm diameter 600 Inconel tubing of 1.067 mm wall thickness. The combustion tube, which is designed to withstand temperatures as high as 1150° C is equipped with heaters and thermocouples; the

heaters are controlled so the tube is able to approach adiabatic conditions. The adiabatic tube is placed in a pressure jacket with a pressure rating of 6,895 kPa.

The following variables are measured throughout a run: pressure drop, temperature along the tube, volume of injected air and water, composition of produced gas, and volumes of produced gas, oil and water. A complete description of the equipment can be found in two other sources (4-5).

EXPERIMENTAL PROCEDURE

A sample of Athabasca oil sands was manually tamped into the tube. Samples were removed every 7.5 to 15.25 cm to determine fluid saturation. After the tube was packed, it was placed in the jacket and brought up to the desired pressure. In order to get the oil to flow it was necessary to pack samples with high water saturation or to heat the combustion tube to about 100°C. After communication through the tube was obtained, the air injection end was heated to about 300°C while flowing nitrogen through the pack. When the ignition temperature was reached, the nitrogen flow was switched to air and the first zone allowed to reach its peak temperature. From this time on, air and water were injected. During the run all of the variables previously mentioned were recorded. The tube was allowed to cool at the end of the run and was then depressured. The tube was removed and unpacked in 7.5 to 15.25 cm sections. Each sample removed was analyzed for fluid saturation and coke content.

GAS ANALYSIS

The produced gas was analyzed on a Hewlett-Packard 5830 three column, dual TC, FID detector gas chromatograph. The gas was analyzed for the following components: oxygen, nitrogen, carbon monoxide, carbon dioxide, hydrogen, methane, ethylene, ethane, propylene, propane, iso-butane, normal butane, iso-pentane, normal pentane, normal hexane, hydrogen sulfide, carbonyl sulfide, and sulfur dioxide.

OIL ANALYSIS

The produced oil and water samples were separated by heating and high speed centrifuging. Samples were heated to 80°C, then centrifuged at speeds of up to 19,500 rpm; heating the sample increased the density difference between the oil and water. Once the oil was separated from the water, the following measurements were performed: weight percent carbon, hydrogen, nitrogen and sulfur; viscosity and density of the oil; weight percent asphaltenes; and a vacuum distillation on the asphaltene free oil.

The weight percent carbon, hydrogen and nitrogen were measured using a Hewlett-Packard Model 185, C, H, N-analyzer. The analyzer oxidized C, H $_2$, and N $_2$ and measured the concentration of these compounds using a thermal conductivity detector. The weight percent sulfur was determined using a Horiba Model SLFA 200 total sulfur analyzer which employs x-ray absorption to measure the sulfur concentration.

Viscosity was measured using a Wells-Brookfield microviscometer which relates shear stress to the torque over a conical surface. Density was measured with a PAAR digital density meter which determines density by measuring the variations of the natural frequency of a hollow oscillator when liquids or gas were introduced into it.

The asphaltene content was determined by taking approximately 10 gms of oil sample, adding 100 ml of pentane and mixing the two at 20°C . The precipitated asphaltenes were filtered out and dried at 100°C . The pentane soluble portion was then distilled into three fractions. First, the pentane and light oil were removed at room temperature by reducing the pressure to 1.2 mm of Hg. The sample was then heated to 200°C at a pressure of 1.2 mm of Hg. The portion distilled off was defined as the middle oil and the residue was defined as the heavy oil. Since many

of the samples were small, it was necessary to develop a special distillation apparatus in which to do the distillations. Figure 2 is a photograph of the device used. A complete description of the procedure and equipment is in a paper by Hayashitani et al. (6). A simulated distillation curve was then obtained on the middle oil using the ASTM-D2887 procedure.

In addition to oil samples obtained from the effluent, samples were obtained from the initial material placed in the combustion tube and from the tube after the burn was completed. The oil was extracted from the sand using the Dean Stark method (7). Following the extraction, toluene was removed by heating the sample to $100^{\circ}\mathrm{C}$ under a pressure of 280 mm of Hg. The type of tests on these samples depended on the amount of sample obtained from the extraction.

WATER ANALYSIS

The water separated from the produced oil was filtered using 20 micron filter paper to remove most of the suspended solids. The following tests were then performed: pH; total organic carbon; potassium, calcium, sodium, magnesium, barium, iron, sulfate, chloride, carbonate and bicarbonate concentrations.

The pH was determined using a Brinkmann Model 104 meter. Total organic carbon was determined using a Beckmann Model 915A total organic carbon analyzer. Operation of the instrument involves injection of aqueous samples into two different combustion tubes using air as the carrier gas. In the total carbon channel, a high temperature (950°C) furnace heats a combustion tube packed with a cobalt oxide-impregnated asbestos fiber. The oxygen in the carrier, the elevated temperature, and the catalytic effect of the packing result in oxidation of both organic and inorganic carbonaceous material to $\rm CO_2$ and steam.

In the inorganic carbon channel, a low temperature (150°C) furnace heats a combustion tube containing quartz chips wetted with 85% phosphoric acid. The acid liberates CO₂ and steam from inorganic carbonates. Operation temperature is sufficiently high for the desired reaction, but is substantially below that required to oxidize organic matter.

The effluent from each combustion tube passes through the associated condenser for removal of condensed steam, and flows to the Sample Select Valve. This valve directs the effluent from a selected channel through a filter to the Model 215A Infrared Analyzer. Here the CO₂ formed during passage of the sample through the combustion tube registers a transient peak on the meter and the recorder chart.

The metal ions concentration was measured using a Perkin Elmer model 303 atomic absorption spectrophotometer, which is equipped to do both flame and flameless analysis.

Sulfates were measured with a standard turbidimetric method. The concentration was determined with a Bausch and Lomb Spectronic 710. Chloride, carbonate, and bicarbonate concentrations were determined by titration. A silver nitrate titration using an Orion solid state chloride specific ion electrode was used for chlorides. The amounts of carbonates and bicarbonates were obtained by titrating 0.02 N, $\rm H_2^{SO_4}$ to a pH end point of between 4.2 to 4.5.

DISCUSSION OF RESULTS

This paper presents the results of two combustion tube experiments. In the first test referred to as Run 1, 1.14 meters of the 1.83 meter tube were burned, and then the tube was cooled. The material was then removed and the oil extracted using the Dean Stark method. In the second run (Run 2) the tube was completely burned through,

and the analysis of the effluent was reported. Tables 1 and 2 present the properties of the material in the combustion tube for these two runs. Table 3 presents the average properties and run conditions. Run 1 was dry while Run 2 was a wet test in the super wet region. Run 1 had a high bitumen saturation so that there would be sufficient oil ahead of the fire-front for the analysis. Run 2 contained a lower concentration for ease of operations with wet combustion. Tables 4 and 5 present the oil analysis data from the two runs. Table 6 and 7 present the simulated distillation data on Runs 1 and 2.

In Run 1 the residual oil analysis data shows a reduction of the light ends, an increase in the asphaltene content of the oil, and an increase in coke content as the fire-front is approached from the production end of the tube. When the fire flood was stopped the front was 1.14 m along the tube. Figure 3 shows the temperature profile at this time. The coke content at this point was 1.84 weight percent. At temperatures above 450°C the oil was all vaporized. Coke started to form at temperatures in the range of 200°C . Ahead of the fire-front the amount of oil gradually increased to a maximum value of 10.25 weight percent at 1.562 m. The data shows that the oxygen content of the crude oil increases significantly in front of the fire front. Some of the low temperature oxidation reactions could have occurred after the fire stopped burning. During the bleed down the air in the tube was passed through this heated oil. The data shows all the oil in the tube had been significantly changed from the initial bitumen. The sulfur content of oil directly ahead of the fire front was reduced.

The analysis of the produced oil from Run 2 shows that the production was gradually upgraded. The weight percent asphaltenes decreased from 20.0 to 5.6 weight percent. The distillable portion increased from 8.6 to 43.7 weight percent. The residue fraction decreased from 71.4 to 50.7 weight percent. The H/C atomic ratio increased from 1.54 to 1.94. The sulfur content decreased from 4.62 to 2.429 weight percent. The viscosity decreased from 400 to 5.5 cp at $80^{\circ}\mathrm{C}$. The density decreased from 1.0155 to 0.9342 gm/cm³ at $25^{\circ}\mathrm{C}$. The simulated distillation data shows that while the amount of the distillable fraction increased with time, the amount distillable at any temperature decreased with time.

Table 8 presents the water analysis data. The pH showed an initial increase from 4.63 to 7.09, then decreased to a low of 1.15. Sodium decreased from 690 to 90 mg/l. Potassium increased from 56 to 270, then decreased to 20 mg/l. Calcium increased from 220 to 340 and then decreased to 26 mg/l before a slight increase. Magnesium decreased from 92 to 18 mg/l. Iron increased from 50 to 1,200 then decreased to 180 and rose to 290 mg/l at the end of the run. Chloride increased from about 180 to 3,500, then decreased for the remainder of the run. Sulfate concentration appeared to increase significantly with a pH below 2. It started out at a value of 3,720, decreased to 230, and then increased to a high of 14,000 mg/l. The pH of the water for most of the run indicated that carbonates and bicarbonates were not present. We believe that the low pH values are caused by Sulfur and CO₂ reactions plus the formation of some organic acids. Sulfate concentration increases indicate that sulfur is reacting to form sulfuric acid. The amount of inorganic carbon from the TOC analysis indicates that there is a possibility of some carbonic acid being present.

The TOC analysis shows hydrocarbon concentrations as high as 7,200 mg/l. We have made no effort to identify the types of hydrocarbon which are dissolved in the produced water. The total dissolved solids ranged from a low of 0.58 to a high of 3.01 weight percent. This curve follows the sulfate curve very closely.

Table 9 presents the average gas analysis for the two runs. Both of these runs show that the only sulfur gas produced was ${\rm H}_2{\rm S}$. Run 2 data indicate that with the injection of water hydrogen was generated.

CONCLUSIONS

From the results of the analysis of the products from these two runs it was concluded that thermal cracking, combustion gas reactions, and hydrogenation caused the following:

- 1. A reduction in the sulfur content of the produced oil.
- An upgraded oil with a higher H/C ratio, lower density, lower viscosity, and lower asphaltene content.
- 3. The sulfur removed from the oil is mainly produced as acid, water, and a small amount of ${\rm H}_2{\rm S}$.
- 4. The produced water contains up to 7,200 mg/l of dissolved organic carbon.

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Sample No.	Mid Point Depth cm.	Weight gms.	Wt. % H ₂ O	Wt. % Bitumen	Wt. H ₂ O gms.	Wt. Bitumen gms.
coarse sand	8.89	3,172.5	6.30	0.0	200.0	0.0
1	24.93	1,475.0	2.20	13.43	32.5	198.1
2	38.58	2,078.0	1.54	12.44	32.0	258.5
3	51.12	1,830.2	2.85	12.86	52.2	235.3
4	62.71	1,619.1	1.15	12.46	18.6	201.7
5	74.22	1,936.0	2.20	12.66	42.6	245.1
6	86.36	1,893.0	1.11	13.11	21.0	248.2
7	99.22	2,110.9	2.90	11.98	61.2	252.9
8	112.72	1,927.3	1.04	11.96	20.0	230.5
9	126.69	1,859.1	1.18	12.86	21.9	239.1
10	139.70	1,760.6	1.57	12.70	27.6	223.6
11	152.88	1,922.8	2.83	11.91	54.4	229.0
12	167.64	2,241.2	2.17	12.17	48.6	272.8
13	178.28	831.0	0.98	12.96	8.1	107.7
coarse sand	181.85	438.6	6.30	0.00	27.6	0.0
TOTAL					668.3	2,743.0

Sample No.	Mid Point Depth cm.	Weight gms.	Wt. % H ₂ O	Wt. % Bitumen	Wt. H ₂ O gms.	Wt. Bitumen gms.
coarse sand 1	8.26 21.12	2,492 1,553	11.0 8.0	0.0 6.5	274.1 136.7	0.0 100.9
2	30.64	1,504.5	9.8	7.0	147.4	105.3
3	40.96	1,611	8.7	6.9	140.2	111.2
4	51.12	1,514	11.7	6.2	177.1	93.9
5	60.17	1,550	10.3	6.7	159.7	103.9
6	79.22	1,539	9.8	6.5	150.8	100.0
7	79.06	1,542	8.9	6.5	137.2	100.2
8	88.43	1,516	10.0	6.9	151.6	104.6
9	98.43	1,553	10.6	7.3	164.6	113.4
10	109.22	1,543	9.7	8.5	149.7	131.2
. 11	119.38	1,493	9.6	6.1	143.3	91.1
12	129.86	1,538	9.5	6.6	146.1	101.5
13	140.97	1,518	9.4	6.6	142.7	100.2
14	152.08	1,554	9.0	5.5	139.9	85.5
15	164.78	2,054	10.0	6.9	205.4	141.7
16	173.83	498	10.0	6.9	49.8	34.4
coarse sand	179.23	1,425	4.0	0.0	57.0	0.0
TOTAL					2,673.3	1,619.0

 $\label{eq:TABLE 3} \mbox{Average Properties and Run Conditions}$

	Run 1	Run 2
Weight of coarse sand - gms.	3,611.1	3,917
Weight of water in coarse sand - gms.	227.6	331.1
Weight of oil sands - gms.	23,484.2	24,080.5
Weight of bitumen in oil sands - gms.	2,743.0	1,619.0
Weight of water in oil sands - gms.	668.3	2,673.3
Weight percent bitumen in oil sands - %	11.68	6.7
Weight percent water in oil sands - %	2.85	11.1
Porosity - %	39.23	39.22
Water Saturation - %	11.7	52.6
Bitumen Saturation - %	48.0	28.3
Run Pressure - K Pa	5,860	5,860
Air Flux - m ³ /m ² -sec	5.95×10^{-3}	9.36×10^{-3}
Water air ratio m ³ /m ³	Dry	3.37×10^{-3}
Initial Properties of Bitumen:		
Density at 25°C gm/cm ³	1.0135	1.0135
Viscosity at 85°C cp	400.00	4,000
Weight percent Carbon	82.91	82.91
Weight percent Hydrogen	10.66	10.66
Weight percent Sulfur	4.62	4.62
Weight percent Nitrogen	0.57	0.57
Weight percent Oxygen by Difference	1.24	1.24
Weight percent Asphaltenes	20.0	20.0
Weight percent Distilled	8.6	8.6
Weight percent Residue	71.4	71.4
Simulated Distillation of Distilled Fraction		
Temperature ^O C	Percent	Distilled

203 0.22 0.22 232 1.54 1.54 245 5.33 5.33 277 16.51 16.51

300 41.95 41.95 323 69.46 69.46 345 84.57 84.57 368 92.61 92.61 391 97.41 97.41 414 100.00 100.00

TABLE 4
Summary of Residual Cil Analysis
Run 1

Position 4 In Tube (M)	Wt-1 Coke	Wt-Z H ₂ 0	Wt-X Bitumen	Wt-3	Wt-X H ₂	M-12 22	Ht-X S	Vc-X Asphaltene	Wr-X Discilled	Vt.Z Residue	Density 8/cc 25°C	Hax. Temp, Zone Reached (°C)	Wt-X Oxygen by difference
1.791	0.0	4.87	7.91	82.90	11.5	0.4	4.144	16.2	17.2	66.6	0.9843	136	1.06
1,715	0.0	7.00	9.37	83.66	11.3	< 0.3	3,725	18.2	17.3	64.5	0,9915	140	1.02
1.638	0.0	5.99	9.05	83.32	11.6	0.5	3.481	21.6	17.8	60.6	1.0064	155	1.10
1.562	2.43	2.00	10.25	78.9	10.7	< 0.3	2.921	27.6	16.1	57.3	1.0610	195	7.48
1.486	5.90	0.69	5.42	71.9	9.6	< 0.3	1.872	41.8	2.9	55.3	1.1102	278	16.63
1,410	3.60	0.69	6.85	77.3	10.3	< 0.3	2.227	35.6	5.6	58.8	1.0894	332	10.17
1,334	4.12	0.57	4.01	82.0	11.0	< 0.3	2.597				1.0339	437	4.40
1,238	4.00		0.4										
1, 137	1.84												
1.054	1.03												

• Measured from air injection end

TABLE 5
Summary of Oil Analysis Run 2

Time hrs.	Density g/cm ³ (25°C)	Viscosity cps (80°C)	Wt-Z C	Wt-I H	Wt-Z N	Wt-% S	Wt-Z Asphaltene	Wt-% Distilled	Wt-I Residue
5.50	0.9543	39.6	82.94	11.6	0.3	4.052			
6.00	0.9754	47.2	82.51	12.1	0.3	3.966	14.6	17.2	68.0
6.50	0.9831	133.6	82.20	12.1	0.3	4.253			••••
7.25	0.9825	98.6			_	4.078	16.7	13.8	69.5
8.00	0.9858	112.6				INS.S.		2310	03.3
8, 25	0.9689	60.95	82.06	12.3	0.5	4.039	15.1	17.0	67.9
8, 50	0.9566	14.8				3.426	11.7	17.3	71.0
9.08	0.9560	19.0	82.92	12,2	0.3	3.432	10,9	26.9	62.2
9.58	0.9564	15.3				3.213		20.7	02.2
10.08	0.9571	17.9				3.359			
10.50	0.9546	16.7				3,257			
10.92	0.9511		82.60	13.0	0.3	2.991	9.4	33.4	57.2
11.58	0.9494	12.3				2.967		33.4	3/.1
12.00	0.9464	· -	82.99	12.5	0.3	3.081	9.1	31.2	59.7
12.25	0.9462	12.0				3.010	···	72.4	33.7
12.95	0.9422					2.718	8.1	27.3	64.6
13.45	0.9402	8.2	82.60	13.3	0.3	2,664	***	-/.5	04.0
13.95	0.9340	_	82.71	13.4	0.3	2.462	5.6	43.7	50.7
14.47	0,9396	8.6			0.2	2.774	3.0	43.7	30.7
14.85	0,9342	5.7				2.429			
15.47	INSUFF.	5.9				INS.S.			
15.92	SAMPLES	5.5				110.01			
16.50	"								
17,20									
17.47	4								

TABLE 6
Simulated Distillation Data
Percent Distilled
Run 1

Temp.		e_* (m)								
Temp. C	1.791	1.715	1.638	1.562	1.486	1.410				
203	1.99	1.17	0.92	0.44	0.40	0.72				
232	11.67	8.95	6.77	3.46	5.74	4.67				
245	29.36	25.47	21.49	13.34	20.58	15.95				
27 7	53.48	50.73	44.70	32.97	42.95	37.04				
305	75.01	74.39	68.41	59.61	68.35	61.97				
323	. 89,41	89.59	86.12	82.54	86.61	81.77				
345	96.16	96.40	95.33	94.34	95.09	93.05				
368	99.06	99.15	99.16	98.8 7	98.77	98.25				
391	100.00	100.00	100.00	100.00	100.00	100.00				
414	100.00	100.00	100.00	100.00	100.00	100.00				

^{*}Measured from air injection end.

TABLE 7
Simulated Distillation Data
Percent Distilled
Run 2

Temp.				Product	ion Time	(Hours)			
Temp. C	6.0	7.25	8.0	8.5	9.08	10.92	12.0	12.95	13.24
203	4.74	3.17	2.75	3.48	1.05	1.13	0.79	0.32	0.38
232	18.56	15.66	14.24	14.90	9.03	8.33	6.59	5.55	4.09
245	13.67	38.06	34.26	33.47	25.38	22.84	20.06	19.01	14.01
27 7	63.36	67.47	59.19	58.00	49.56	44.69	41.74	41.42	30.68
300	81.47	86.67	78.62	79.97	73.47	68.98	66.48	65.93	54.68
323	92.16	95.15	90.81	92.41	89.07	86.95	85.50	84.47	79.08
345	97.13	98.22	96.80	97.47	96.34	95.89	95.27	94.57	93.53
368	99.26	99.82	99.20	99.37	99.05	99.24	98.89	98.52	98.19
391	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
414	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00

TABLE 8

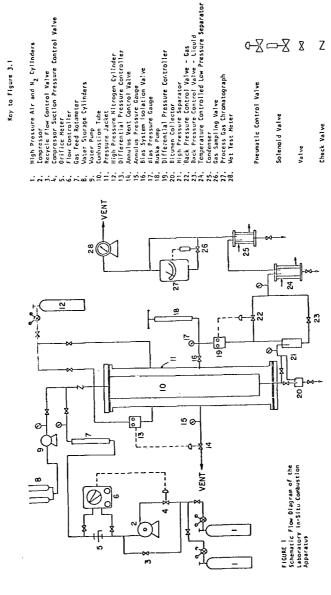
Summary of Water Analysis - Run 2

Total	Solids %	0.72	0.61	0.00	0.68	0.71	0.58	0.58	0.84	1,05	1.07	1.14	0.99	0.84	96.0	1.16	1.33	1.48	1.33	1.27	1.27	1,35	1.48	1.51	3.01	2.44	2.69	2,14	1,60	1,94	1.51	1.10	1.00	0.82
	Organic Carbon	3,735	867)								2.882					5.133	21-6-											7,232					1,938
TOC	inorg. Carbon	15	13	1								18					17	i											18					12
	Total	3,750	077									2,900					5,150												7,250					1,950
HCO F	т / 8ш																			-														
C0 32-	T/Sm																																	
S0,2-	т/8ш	3,720	2,880	2,000	1,520	780	240	200	480	420	270	230	330	320	1,220	2,770	3,610	4,560	4,180	4,480	4,980	5,920	6,180	094,9	14,000	10,700	12,300	9,850	7,100	8,280	6,860	5,370	4,870	3,400
ដ	1 / Sim	230	180	300	770	1,100	1,000	1,300	2,000	2,900	3,700	4,700	3,500	3,100	2,500	2,400	2,100	1,700	1,500	1,300	400	260	610	400	260	1,100	110	< 15	< 15	< 15	< 15	< 15	< 15	< 15
Fe 7	11/Sim	180	50 42	250	330	480	200	670	790	1,100	000° T	1,100	900	720	240				250					180	430				290		290	220	240	
Ba	11 / SIII	,		· -	٦ ٧	V	1	^	۲ ۷	Ţ. •	е н У	, ,	٦ ٧	,					٦ ٧					- '	п v						٦ ٧	, ,	⊢ v	
Mg	1867	92	85	80	87	29	62	70	78	92	78	61	20	38	32				30					28	63				20		19	18	18	
Ca 7	184	220	220	220	230	240	200	230	290	340	300	250	200	160	93				30					56	26				35		70	43	77	
X K	÷ /9;;	56	92	81	95	110	100	130	160	160	230	260	220	230	270				160					62	89				53		32	23	7.7	
Na mo/1	i .	069	610 530	410	350	200	130	120	130	100	80	99	9	53	43				84					11	17				11		10	13	70	
Нď		4.63	7.09	4.10	3,51	3.39	3.51	3,54	3.20	2.93	2.24	1,66	1,69	1,73	1.54	1.49	1,45	1,43	1.49	1.49	1,48	1.45	1,41	1.41	1.13	1,17	1.15	1.20	1,30	1.26	1.43	1.64	T. /6	7.03
Time		1.10	2.95	3,92	4.28	4.67	5.00	5.50	9.00	6.50	7.25	8.00	8.25	05°	80°6	9.58	10.08	10.50	10.92	11.58	12.00	12.25	12.95	13.45	73.95	14.47	14.85	15.47	15.92	16.50	17.20	17.47	10.08	LC . 47

TABLE 9

Gas Analysis

	Run 1	Run 2
	Mole-%	Mole-%
	0.5	
Nitrogen	86.5	87.06
Oxygen	0.0	0.08
Carbon Dioxide	10.60	11.47
Carbon Monoxide	1.93	1.34
Methane	0.62	0.02
Ethane	0.05	0.00
Ethylene	0.05	0.00
Propane	0.02	0.00
Propylene	0.01	0.00
C ⁴ ÷	0.20	0.00
Hydrogen Sulfide	0.02	0.01
Hydrogen	0.00	0.02



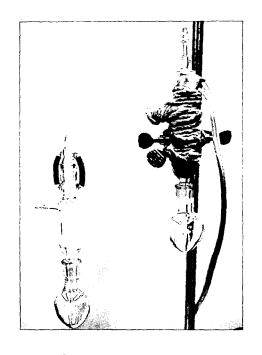


FIGURE 2
Distillation Apparatus

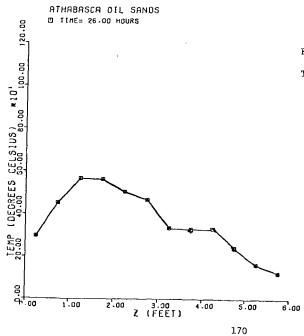


FIGURE 3
Temperature Profile